(19)

## PATENT SPECIFICATION

352 200

(21) Application No. 16007/74

(22) Filed 10 April 1974

(31) Convention Application No's 351 793 (32) Filed 16 April 1973 18 April 1973 in

(33) United States of America (US)

(44) Complete Specification published 17 Nov. 1976

(51) INT. CL.2 F04B 43/06 G01N 1/14

(52) Index at acceptance

FIR 11 15A 3A3A

B1X2

GIB 13A4

## (54) A FLUID METERING DISPLACEMENT DEVICE, METHOD AND SYSTEM

We, COULTER ELECTRONICS, INC., an Illinois Corporation, of 590 West 20th Street, Hialeah, Florida, United States of America, do hereby declare the invention, 5 for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:-

The embodied methods, device and system relate to precision movement of

small volumes of fluid.

Generally speaking, the movement of small volumes of fluid, for example liquid 15 chemicals and reagents in analysis arrangements, has been accomplished by several forms of devices and systems having as a common goal the precise reproducibility of the volume movement, i.e. displacement.

The embodiments hereinafter disclosed also have the goal of volume displacement reproducibility with the use of a displacement device having a cavity in which there is a diaphragm which is pressure driven 25 between two opposite stable conditions,

which enables the volume of the cavity to define the desired volume displacement. The primarily embodied diaphragm does not have to be of an elastomeric material 30 and can be inert to a large range of

chemicals.

In a fluid displacement system, it is desirable that a minimum of crosscontamination between samples be incurred.

35 Accordingly, a method and system of sampling a liquid utilizing the fluid displacement device hereinafter to be described in detail, provides for a minimum of crosscontamination between liquid samples.

According to the invention there is provided a method of making a fluid displacement device which has a substantially constant volume displacement, said method comprising the steps of: deforming a

45 flexible inelastic diaphragm to cause it to

have substantially the same shape and surface area as the shape and surface area of each concave surface of a hollow body of fixed volume with oppositely facing. interior, similarly shaped, concave sur- 50 faces having substantially equal surface areas; and then clamping the periphery of the diaphragm between the peripheries of the concave surfaces, and thereby mounting the diaphragm for movement between and 55 against the concave surfaces with the area of the diaphragm within the space defined by the periphery of each concave surface of the hollow body being greater than the planar area defined by the periphery of each 60 concave surface; diaphragm movement from one surface to the other surface defining the fixed volume to be displaced. According to the invention there further

is provided a fluid displacement device 65 made in accordance with the just above set

forth method.

According to the present invention there further is provided a method for utilizing a fluid displacement device of the type 70 described in the immediately preceding paragraph, the device being coupled to a sampling element for sampling a precise amount of a sample material from a source thereof, comprising the steps of: placing 75 the diaphragm in a predetermined first position intermediate the two concave surfaces of the hollow body of the device, the diaphragm and a first of the concave surfaces defining therebetween a pre-80 determined first volume which is significantly less than the total displacement volume of the device; the sampling element being in fluid connection with the interior of the displacement device by way of its 85 second concave surface, and the sampling element being in contact with the sample material, moving the diaphragm to a second position conforming against the first concave surface and thereby drawing into 90

the sampling element a first volume of the sample material equal to said first defined volume; separating the sampling element from the source of sample and moving the 5 diaphragm to a third position conforming

against the second of the concave surfaces and thereby dispensing from the sampling element a total volume equal to the total displacement volume of the device and thus 10 all of said first volume of the sample

material and an additional volume of substance for cleaning the sampling element prior to a next cycle of sampling by this

The preferred embodiments of this invention now will be described, by way of example, with reference to the drawings accompanying this specification in which:

Figure 1 is a front perspective view of a 20 fluid displacement device;

Figure 2 is an exploded perspective view of the device shown in Figure 1;

Figure 3 is a sectional view taken through

the device along the line 3-3 of Figure 1 25 and in the general direction indicated; Figure 4 is a side view partially schematic

and with portions broken away of one embodiment of a liquid sampling system embodying the device of Figure 1; and

Figures 5A-5D are side views partially schematic and with portions broken away of another embodiment of the liquid sampling system of Figure 4 showing different stages of operation thereof.

Referring to Figure 1, the embodied fluid displacement device 10 has three principal parts. Two of the parts are a pair of mating half shells 12, 14 which can be injectionmoulded and, preferably are substantially

40 identical and possess annular flanges 19. The third part is a flexible diaphragm 16 sandwiched between the two shell halves 12 and 14 and held in place by fasteners engaged through registering openings in the 45 annular flanges.

Each shell has a nipple 22, 24 which is connectable to conduits 26, 28 for connecting the fluid displacement device into a fluid system, such as shown in Figure 4.

Referring to Figure 2, the shells 12, 14 have chambers 30 and 32 each bordered by its flange 19. Each of the flanges 19 includes an annular clamping ring 41, 42 having the diaphragm 16 sandwiched therebetween.

Diaphragm 16 is clamped between the shells in such a manner that the total area of the diaphragm within the clamping rings 41, 42 is greater than the central planar area defined by the annular ring by 60 an amount sufficient for the diaphragm to be able to conform with and alternately lie against the interior surface of the chambers 30, 32. During construction the diaphragm is assembled and pressure is applied to 65 drive the diaphragm into engagement and

conform with the inner surface of one chamber of one shell. While in this condition, the shells are permanently fastened together. Thereafter, the diaphragm can be moved by fluid pressure differentials from 70 one to the other interior surface of the device in a toggle-like action, with little or

no stretching of the diaphragm.

The term "toggle-like action" is employed herein to designate the property of 75 the diaphragm and the operation of the device 10, which provides two stable state positions, as shown in Figure 3 as 16 and 16', both conforming with the inner wall of the chambers 30 and 32. Suitable differ- 80 ential pressure is employed to drive, by pushing or pulling, the diaphragm through its "toggle-like action."

Hence, the diaphragm is of a material which is not elastomeric. For example, the 85 diaphragm can be made of polytetrafluoroethylene which also is quite inert.

Each of the shells has an antechamber 46, 48 interposed between the nipples 22, 24 and the chambers 30, 32. The ante- 90 chambers 46 and 48 enable sufficient differential pressure to be applied to either side of the diaphragm 16 to cause the diaphragm to toggle back and forth.

When the diaphragm is being moved into 95 position 16', fluid will be forced out of the chamber 32 via the antechamber 46 through the nipple 24, and fluid will be forced into the chamber 30 via the antechamber 48 through the port 42 from tube 100 26 coupled thereto.

The fluid volume displaced by the toggle-like action of the diaphragm 16 will not change with time, since the volume is dependant only upon the fixed volume of 105 the chambers 30 and 32; hence, an accurately reproducable volume displacement of fluid is achieved by the subject device 10.

A liquid sampling system utilizing the fluid displacement device 10 of Figures 1-3 110 generally is indicated by the reference numeral 110 in Figure 4. The system 110 includes a sampling element, for example a cuvette 112, the fluid displacement device 10, a fluid connection line 116 connecting 115 the device 10 with the upper end 117 of the cuvette 112, and a line 152 between the device 10 and a control valve arrangement 118 for controlling the operation of the device 10. The arrangement of the ante- 120 chambers 46, 48 and nipples 22, 24 are not shown in Figure 4, for simplicity of drawing. The valve arrangement 118 includes a control valve 154 and a throttling device 156 in the form of a needle valve. 125 As shown, a fluid line 158 leading to a source of vacuum and a fluid line 160 leading to a source of pressure are connected to the valve 154.

In the illustrated embodiment of the 130

liquid sampling system 110 shown in Figure 4, an air line 162 with a valve 164 therein is connected into the fluid line 116 between the displacement device 10 and the cuvette 5 112. The valve 164 is operable to communicate pressurised air through the fluid line 116 to the upper end 117 of the cuvette 112.

The method of using the system 110 is initiated by operating the valve 154 to 10 connect the pressure line 160 through the throttling device 156 to the volume displacement device 10 to push the diaphragm 16 upward against the inner surface of the cavity 30. Then, the lower end 122 of the 15 cuvette 112 is placed into a body of sample liquid 166, as in a test tube 168. Next, the valve 154 is operated to connect the vacuum line 158 to the device 10 to pull the diaphragm downwardly to the position 20 shown in Figure 4. In this way, a precise

quantity of fluid is drawn up from the body of liquid 166 into the cuvette 112. The needle valve 156 throttles the suction applied to the device 10 so that the 25 diaphragm 16 can move slowly from the

surface of the cavity 30 to the surface of the cavity 32. As a result, the liquid drawn into the cuvette 112 is drawn thereinto slowly and smoothly without splashing of

30 the liquid into the upper end 117 of the cuvette 112.

Although the sampling element 112 is not a limitation upon the scope of the invention, the cuvette 112 and photometric elements 35 130 and 132 are described as a practical example. A photometric analysis now can be made by passing light through the cuvette from the source 130 to the photosensitive device 132. Before or after the

40 photometric analysis, the lower end 122 of the cuvette 112 is removed from the body of liquid 166. Then, after the photometric analysis-has-been-made, the valve 154 is operated again to connect the pressure line

45 160 to the volume displacement device 10, to move the diaphragm 16 against the inner surface of the cavity 30 and thereby force the liquid out of the cuvette 112. Next, with the lower end of the cuvette 122 open to

50 the ambient air above a waste container (not shown), the valve 154 is operated to connect the vacuum line 158 to device 10 to draw a quantity of air into the cuvette 112 after which the valve 154 is connected to

55 the pressure line 160 to operate the device 10 to force the air out of the cuvette 112 and in so doing to eject from the cuvette any droplets of liquid which may have clung to the interior side walls of the 60 cuvette.

As a modification to the method of operaing the liquid sampling system described above, after the liquid sample is ejected from the cuvette 112, the cuvette 112 can 65 be lowered into a source of rinse liquid

which is then drawn up into the cuvette and subsequently purged from the cuvette prior to the drawing of air into the cuvette. This is accomplished by operating the valve 154 in the manner described above to operate 70 the volume displacement device 10 in the manner described above.

Also, and to save time to provide for a better blowing out of liquid from the cuvette 112, the step of pushing air through the 75 cuvette 112 can be performed or assisted by operating the valve 164 to transmit pressurized air in the line 162 through the fluid line 116 to the interior of the cuvette 112. With this modification, the device 10 need 80 not be operated to pull air into and then force air out of the curvette 112.

Turning now to Figures 5A-5D, another method of operating the embodied liquid sampling is shown. With appropriate pres- 85 sure differential being applied to the diaphram 16, it will lie in a neutral position between the shells 12, 14 as shown in Figure 5A. While the diaphram 16 is shown as being flat in the neutral position, 90 this of course is not actually possible since the area of the diaphram is greater than the planar area of the cavity in each shell. The diaphram will actually be wrinkled.

The valve arrangement 218 is operable to 95 connect the cavity 30 to either a line 244 leading to ambient air, a line 246 leading to a source of vacuum or to a line 248 leading to a source of pressure. If desired, a connecting line 250 of the valve arrange- 100 ment 218 can include a throttling device similar to the needle valve 156 shown in

Figure 4.

In the method of utilizing the liquid sampling system and assuming the neutral 105 diaphram position divides the volume defined by the cavities 30 and 32 in half, the valve arrangement 218 is operated to connect the cavity 30-to-the ambient air line 244 so that the diaphram 16 is in a neutral 110 position with half of the volume capacity of the device 10 on either side of the diaphram and with air in the cavity 32. It is understood that the diaphram 16 can have a neutral position which is not necessarily at 115 the midpoint of the chamber defined by the cavities 30 and 32. Then the lower end 122 of the cuvette is lowered into the body of liquid 166. Subsequently, the valve arrangement 218 is operated to connect the 120 cavity 30 to the vacuum line 246 as shown in Figure 5B. This results in a volume displacement of one-half the volume capacity of the device 10 to draw that quantity of the liquid in the container 168 upwardly 125 into the cuvette 112. After a photometric analysis of the sample in the cuvette 112 is made and after the cuvette is removed from the container 168 and placed over a waste fluid receptacle 260 shown in Figure 5C, 130

the valve arrangement 218 is operated to communicate the cavity 30 with the pressure line 248. The diaphram 16 thus is moved completely across the device 10 from the 5 inner surface of the cavity 30 to the inner surface of the cavity 32. In this way, not only is the fluid in the cuvette 112 forced out of the cuvette 112, but also a quantity of air, which had been stored in the cavity 10 32 and which is equal to one-half the volume displacement of the device 10, is forced out of the cuvette. This quantity of air serves to remove droplets of the sample liquid which may have clung to the interior 15 side walls of the cuvette 112. Subsequently, and as shown in Figure 5D, the valve arrangement 218 is operated to connect again the ambient air line 244 to the cavity 30 so that the diaphram 16 is returned to its 20 neutral position and a quantity of air equal to one-half the volume displacement of the device 10 is drawn into the cavity 32. The system now is ready for the sampling of another liquid sample from a next con-25 tainer.

From the foregoing description it will be apparent that the embodied displacement device, liquid sampling system and the methods of liquid sampling described pro-30 vide for a very efficient and precise sampling of liquid with little or no cross contamination between samplings of liquid.

WHAT WE CLAIM IS

1. A method of making a fluid displace-35 ment device which has a substantially constant volume displacement, said method comprising the steps of: deformflexible ing а inelastic diaphram to cause it to have substantially 40 the same shape and surface area as the shape and surface area of each concave surface of a hollow body of fixed volume with oppositely facing, interior, similarly shaped, concave surfaces having 45 substantially equal surface areas; and then clamping the periphery of the diaphram between the peripheries of the concave surfaces, and thereby mounting the diaphram for movement between and against the con-50 cave surfaces with the area of the diaphram within the space defined by the periphery of each concave surface of the hollow body being greater than the planar area defined by the periphery of each concave surface: 55 diaphram movement from one surface to the other surface defining the fixed volume to be displaced.

2. The method according to claim 1 wherein the step of deforming comprises the 60 steps of: holding the periphery of the diaphragm between the concave surfaces; effecting a pressure differential on opposite sides of the diaphram such that the diaphragm deforms to conform to one of the 65 concave surfaces.

3. The method according to claim 2 wherein the pressure differential is obtained by applying fluid pressure to one side and vacuum to the other side of the diaphragm.

4. A fluid displacement device made in 70 accordance with the method of any one of claims 1, 2 or 3.

5. The fluid displacement device according to claim 4 including: fluid ports opening through the concave surfaces for esta- 75 blishing pressure differential on opposite sides of the diaphragm for effecting diaphragm movement.

6. The fluid displacement device according to claim 4 or 5 including an annular 80 ring on the periphery of each of the concave surfaces for holding the periphery of the deformed diaphram therebetween.

7. The fluid displacement device according to claim 6 wherein said diaphram and 85 said annular rings have a substantially cir-

cular periphery.

8. The fluid displacement device according to any one of claims 4 to 7 wherein said diaphram is of a chemically inert 90 material.

9. The fluid displacement device according to any one of claims 4 to 8 wherein said diaphragm is polytetrafluoroethylene.

10. The fluid displacement device accord- 95 ing to any one of claims 5 to 9 wherein each of the concave surfaces includes an antechamber interposed between the interior of the device and one of said ports.

11. The fluid displacement device accord- 100 ing to any one of claims 5 to 10 wherein a sampling element is coupled to one of said ports and pressure differential means are coupled to another port of coupling alternately to said device a source of pres- 105 sure and vacuum such that said device draws liquid into and pushes liquid out of said sampling element when pressure differentials are created on both sides of said diaphragm.

12. The fluid displacement device according to claim 11 wherein said pressure differential means include throttling means for throttling the magnitude of the differential pressure created on both sides of said dia- 115 phragm to cause said diaphragm to move slowly from one surface to the other thereby causing liquid to be drawn into and pushed from said sampling element smoothly and slowly.

13. A method for utilizing a fluid displacement device according to any one of claims 4 to 12, the device being coupled to a sampling element for sampling a precise amount of a sample material from a source 125 thereof, comprising the steps of: placing the diaphragm in a predetermined first position intermediate the two concave surfaces of the hollow body of the device, the diaphragm and a first of the concave surfaces 130

defining therebetween a predetermined first volume which is significantly less than the total displacement volume of the device; the sampling element being in fluid continuous continuous

5 nection with the interior of the displacement device by way of its second concave surfaces, and the sampling element being in contact with the sample material; moving the diaphragm to a second position con-

10 forming against the first concave surface and thereby drawing into the sampling element a first volume of the sample material equal to said first defined volume; separating the sampling element from the

source of sample and moving the diaphragm to a third position conforming against the second of the concave surfaces and thereby dispensing from the sampling element a total volume equal to the total displace-

20 ment volume of the device and thus all of the said first volume of the sample material and an additional volume of substance, for cleaning the sampling element prior to a next cycle of sampling by this method.

25 14. The method according to claim 13 in which said first diaphragm position is attainable repeatedly in each of a plurality of subsequent cycles according to this method and the said moving to all of the

positions is accomplished by applying dif- 30 ferential pressures to the diaphragm.

15. The method according to claim 14 including the applying of fluid pressure and vacuum to the side of the diaphragm facing the first concave surface for obtaining the 35 differential pressures.

16. The method according to any one of claims 13 to 15 including the step of throttling the moving of the diaphragh to draw and dispense smoothly and slowly the 40 sample material into and from the sampling

element.

17. The method for making a fluid displacement device substantially as herein described with reference to Figures 1, 2, 45 and 3

and 3.

18. A fluid displacement device, substantially as herein described with reference to Figures 1 to 4.

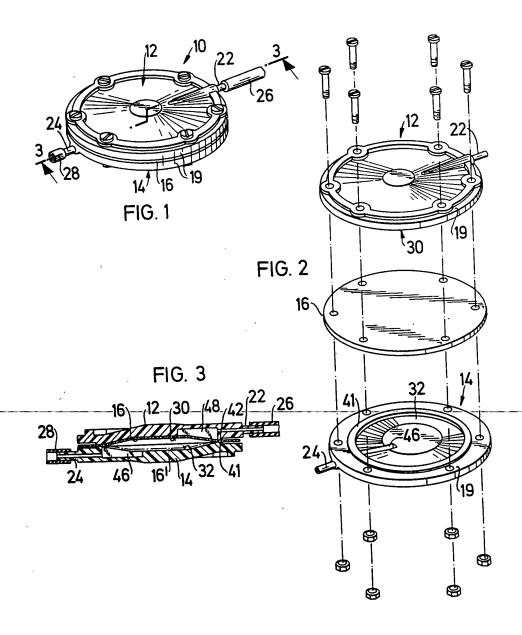
19. A method utilizing a fluid displace- 50 ment device, substantially as herein described with reference to Figure 5.

A. A. THORNTON & Co. Chartered Patent Agents, Northumberland House, 303/306 High Holborn, London, WC1.

Printed for Her Majesty's Stationery Office by The Tweeddale Press Ltd., Berwick-upon-Tweed, 1976.

Published at the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.





. 4 

•

1 455 664 COMPLETE SPECIFICATION

4 SHEETS This drawing is a reproduction of the Original on a reduced scale.

SHEET 2

